

RESEARCH

PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improvements in or relating to Carbon-Monoxide Detecting Substances and Methods of Making the same

1. COLLIS DORE, a Citizen of the United States of America, of 111, West Cypress Street, Santa Maria, California, United States of America, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The present invention has reference to a substance for use in showing the presence of carbon monoxide in a gas mixture (e.g. the atmospheric air in a mine or a room or a compartment) and 15 for producing a suitable strip useful for such showing. In a preferred modification, the material and the strip can be used for determining, at least approximately, the relative amount of carbon 20 monoxide in the atmosphere.

The present invention provides a carbon monoxide colorimetric detector substance comprising an absorbent carrier having deposited on the surface thereof a mixture of a palladium halide and ortho tolidine or sodium alizarin sulphonate.

The present invention also provides a method of making a carbon monoxide colorimetric detector substance which 30 comprises depositing on the surface of an absorbent carrier a mixture of a palladium halide and ortho tolidine or sodium alizarin sulphonate from a liquid and baking the resulting substance.

35 The present invention further provides a carbon monoxide detector strip for showing the presence of carbon monoxide in a gas mixture comprising a strip of material carrying on its surface a substantially uniform layer of said detector substance.

It is well known that small amounts of carbon monoxide in the atmosphere render the same unsuitable for breathing.

40 By the use of the detector substance of the present invention it is possible to demonstrate the presence of as little as 0.002%, or 2 parts in 100,000 parts of air

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(herein, as is common, parts of carbon monoxide in the atmosphere are stated by volume).

In making my composition, the same will be manufactured in an atmosphere which is free from carbon monoxide, and the strip, made up into a roll if desired, 55 can be transported in an air-tight receptacle to the place of use, say a mine, a tunnel through which automobiles are passing, or the like.

Preferably when preparing my detector 60 substance an acidulated solution of a palladium halide is treated with a solution of the ortho tolidine or sodium alizarin sulphonate, the resulting solution treated with a finely-divided adsorbent 65 which is sufficiently transparent to allow light to pass therethrough, and the adsorbent material dried with the products it has picked up from the solution and then baked. After this the material 70

may be distributed upon the adhesive side of a transparent strip of material, in an even, uniform manner, after which the strip of material can be rolled up, placed into an air-tight container and sealed 75 against air containing carbon monoxide.

In the preferred modification the palladium halide used is palladium chloride while sodium alizarin sulphonate (commonly known as alizarin red S) is used 80 rather than ortho tolidine. The adsorbent material preferred is silica gel, preferably of about 14 to 20 mesh, although other fine mesh gels can be used as a substitute.

The most satisfactory strip base 85 which I have experimented with is the transparent tape coated on one side with a pressure sensitive adhesive, known to the trade as Scotch tape, e.g. that known as number 107, of the widths of about three-quarters inch up to one and one-quarter inch. This already carries a satisfactory adhesive on one face, and the said strip is of a high degree of transparency.

For more completely explaining the 95 nature of the invention, are given the

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following Examples, to which the invention is not limited. The examples given represent small scale manufacture on an experimental scale, for the production of small quantities of the products for testing purposes.

EXAMPLE 1.

100 minimis of triple distilled water were placed in a borosilicate glass beaker, 10 and 2 minimis of nitric acid were added, and the liquid well stirred. To the above, 17 minimis of palladium chloride 5% solution were added. Then another 50 minimis of distilled water were added. 15 This mixture was well stirred.

To the above solution 8.5 minimis of a 1% solution of sodium alizarin sulphonate (alizarin red S) was added, and this was added drop by drop, the solution being 20 well agitated while the dye solution was being added.

To the liquid was then added 133 grains of silica gel of about 20 mesh. This was the grade known as Pa 100, made by 25 Davison Chemical Company. The mixture was agitated and allowed to stand for the silica gel to adsorb whatever it would take up from the liquid.

The impregnated gel was then placed 30 in a borosilicate glass evaporating dish and placed under the heat rays of a G.E. reflector Infrared Industrial 250 Watt lamp. The lamp was placed 17 inches above the evaporating dish, and the heat 35 rays and infra red rays were projected directly downwardly into the said dish. The temperature at this stage, just above the gel in the evaporating dish was between 110° F. and 120° F. The gel 40 was constantly stirred up until dry enough to run freely in the dish, as a powder.

The gel was then removed from the lamp, and allowed to cool for an hour. 45 The dish containing the gel was then put into an electric oven and heated for 30 minutes at 290° F. The oven was held at substantially constant temperature.

The dish of gel was removed from the 50 oven and allowed to cool gradually in an uncontaminated atmosphere, to room temperature of 70° F. The gel was then placed into an airtight container and was allowed to stand for between 24 and 48

55 hours.

The dried gel, which was in the form of a relatively fine powder was distributed over the adhesive surface of a strip of Scotch tape (the grade known as No. 107) 60 was used. The gel was worked over the adhesive surface, and finely pressed and rolled firmly into place in the adhesive. The excess of gel was collected on a clean paper, the dried gel on the adhesive surface being brushed with a soft brush to

remove the excess of loose particles.

In other experiments the above formula is varied as follows:—

EXAMPLE 2.

Instead of 17 minimis of 5% palladium chloride, various amounts between 15 and 30 minimis were used.

EXAMPLE 3.

In place of 50 minimis of distilled water added after the palladium chloride solution, various amounts between 25 and 100 minimis were used. These variations in the amount of water (variations in the concentrations) appear to regulate the sensitivity of the compound.

EXAMPLE 4.

Instead of the 1% solution of sodium alizarin sulphonate a solution of ortho tolidine (c.p.) was used. This solution was made up by adding 20 minimis of 10% hydrochloric acid, plus 10 minimis of water, plus 1 grain of ortho tolidine powder. The amount of the ortho tolidine solution employed may vary between 3 and 7 minimis, 5 minimis being a very satisfactory amount.

In the examples given the solution, after adding the alizarin sulphonate, did not appear to be colloidal. During the treatment of the solution after adding the dyestuff with silica gel, the odour of chlorine was noticeable. When the material was placed in the electric oven and heated to 290° F., a lot of chlorine is given off.

The colour of the gel directly after adsorbing the solution, (when being placed into the evaporating dish) was reddish yellow. The heat from the lamp caused the colour to change to a pale gold 105 colour, which colour was retained during the heat treatment in the electric oven.

If ortho tolidine solution is used in place of sodium alizarin sulphonate, the colour before being placed under the lamp is brick red. After drying under the lamp it becomes a slightly lighter red colour. After treating in the electric oven at 290° F., it remains light red, with a purple tinge.

By use of the tape or strip, made in accordance with Example 1 it was readily possible to demonstrate the presence of 0.005% of carbon monoxide. For demonstration gas mixtures containing known amounts of carbon monoxide gas, mixed with pure nitrogen were used. In these tests the rate of flow of the gas against the indicating tape was one pound per square inch, for one minute, the gas 120 coming through a laboratory hose or tube of one-quarter inch diameter.

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On testing an atmosphere containing 0.005% of carbon monoxide, a drop of 10 microampères in the electric circuit of a selenium cell illuminated through the strip was noted, equivalent to a drop of 2.5 foot candle power. In some tests the same part of the strip carrying the chemical was used several times, and after the first treatment, there was no further 10 changes in the reading. Thus the tape is "non-accumulative".

It was found that atmospheres containing 2, 3 and 4 times the above stated amount of carbon monoxide, gave strictly 15 proportional drops in the microampères or foot candles.

On testing with a gas mixture containing a heavy concentration of carbon monoxide (about .1%) the tape immediately turned completely black.

In a test with a 0.01% CO mixture, the colour of the coating on the Scotch tape changed from a pale golden colour to a darker gold colour with a purple tinge. In 25 testing a 0.015% mixture, the purple colour became more apparent. A tape tested with an 0.02% carbon monoxide mixture became a distinct purplish colour. Testing the tape with a gas containing a 30 heavy concentration of carbon monoxide, (about .12%) the tape became purplish black in colour. Tape made according to Example 4 changed from light red to black on exposure to carbon monoxide.

35 Other gases present in the atmospheres tested did not seriously interfere with the use of the tape. In a particular instance, the tape was first treated with hydrogen sulphide gas of a relatively heavy concentration (about .25%). This gas affected the colour of the tape slightly, but after that the 0.01% CO gas treatment was tried on this same tape and found to give a practically correct 45 reading.

Gas containing air and considerable formaldehyde vapour, and also air containing considerable steam vapour had little or no effect on the tape. The tape 50 was thereafter tested with an 0.01% CO gas and functioned perfectly. Also both of these tapes turned completely black from the heavy concentrations of CO gas (about .25%).

55 What we claim is:—

1. A carbon monoxide colorimetric detector substance comprising an absorbent carrier having deposited on the sur-

face thereof a mixture of a palladium halide and ortho tolidine or sodium 60 alizarin sulphonate.

2. A detector substance according to claim 1 wherein said palladium halide is palladium chloride.

3. A detector substance according to 65 claim 1 wherein said absorbent carrier is silica gel.

4. A carbon monoxide colorimetric detector strip for showing the presence of carbon monoxide in a gas mixture comprising a strip of sheet material carrying on its surface, a substantially uniform layer of a finely divided carbon monoxide detector substance, according to claim 1, 70 2 or 3.

5. A method of making a carbon monoxide colorimetric detector substance which comprises depositing on the surface of an absorbent carrier a mixture of a palladium halide and ortho tolidine or 80 sodium alizarin sulphonate from a liquid and baking the resulting substance.

6. A method according to claim 5 wherein said liquid is an acidified aqueous solution and said carrier is silica gel. 85

7. A method of making a carbon monoxide colorimetric detector substance which comprises treating an absorbent carrier with an aqueous acid solution of equal volumes of 5% palladium chloride 90 and 1% sodium alizarin sulphonate to deposit a mixture of the palladium chloride and sodium alizarin sulphonate on the surface of the carrier, drying the resulting product at a temperature 95 between 110° F. and 135° F. and thereafter baking the resulting product for about half an hour at a temperature between 280° F. to 350° F.

8. A carbon monoxide colorimetric 100 detector substance substantially as herein described with particular reference to the Examples.

9. A carbon monoxide colorimetric detector strip substantially as herein 105 described.

10. A method of making a carbon monoxide colorimetric detector substance substantially as herein described.

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